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COMMONWEALTH OF AUSTRALIA

Patent Act

COMPLETE SPECIFICATION

(ORIGINAL)

26281 m

Section 41

Class

Int. Class

Application Number:

Lodged:

Complete Specification Lodged:

Accepted:

Published:

Priority:

Related Art:

PATR

APPLICANT'S REF.: DS48244A

Name(s) of Applicant(s):

N.Z. FOREST PRODUCTS LIMITED.

Address(es) of Applicant(s):

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JOHN WILLIAM WHEATON.

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Melbourne, Australia, 3000

Complete Specification for the invention entitled:

**"IMPROVEMENTS IN AND RELATING TO THE PROCESSING
OF MATERIALS"**

The following statement is a full description of this invention, including the best method of performing

the invention:

1120 PATENT ABSTRACT
1121 AB

(11) AB-61-26 281/77

(54)	BARK EXTRACTION		
(71)	M. I. FOREST PRODUCTS LIMITED		
(21)	28 281/77	518 703	(22) 21.6.77
(23)	21.6.77		(24) 24.6.76
(31)	181274	(32) 24.6.76	(33) NZ
(43)	4.1.79		(44) 15.10.81
(51)	C07G 17/00		
(72)	Swan, D., ET AL		
(74)	P0		
(56)	28 884/77	517 828	C07G
	49 969/59	242 409	09.9

(57) The term "an aqueous, phenolic, bark extract" as used throughout the specification and claims is hereinafter defined as an aqueous solution including at least some phenol or phenolic material, having been extracted from bark, such as for example bark from trees or shrubs.

Claim 1. A method of treating an aqueous, phenolic bark extract including:

adjusting the pH of said extract so that the pH is above pH 7;

maintaining said extract temperature above 49°C for a period of up to 90 minutes;

adjusting the pH of said extract to below pH 7.

20281 77

of 11 6, Roke Road, Penrose, Auckland, New Zealand.

hereby apply for the grant of a Patent for an invention entitled

"IMPROVEMENTS IN AND RELATING TO THE
PROCESSING OF MATERIALS"

which is described in the accompanying (a) complete specification.

(Note: The following paragraph applies only to Convention applications)

This application is a Convention application based on the basic application(s) for a patent or similar protection identified by number, country, and filing date as follows:

181,274

New Zealand.

24th June, 1976.

21 JUN 1977

FOR STAMPS TO VALUE OF £ 72
ATTACHED
MAIL OFFICE

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Dated on 20th June, 1977.

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INVENTION OF A PATENT

Form 211

INVENTION OF A PATENT

In support of the Convention application made by

N.Z. FOREST PRODUCTS LIMITED

(hereinafter called "applicant(s)") for a patent in
invention entitled as

26281

for an

"IMPROVEMENTS IN AND RELATING TO THE
PROCESSING OF MATERIALS"

1/24 =

DAVID FULTON FOWLES
15 PRILEY ST
HOWICK.

do solemnly and sincerely declare as follows:

1. I am/we are the applicant(s).

(or, in the case of an application by a body corporate)

1. I am/we are authorized to make this declaration on behalf of the applicant(s).

2. I am/we are the actual inventor(s) of the invention.

(or, where the applicant(s) is/are not the actual inventor(s))

2. ^{as} David Alan Sugg,
Chick Ing Chiang,
Stephen John Grace,
John Peter Leader and
John William Wheaton, all care of O'Shea Road,
Penrose, Auckland, New Zealand.

I/we the actual inventor(s) of the invention and the facts upon which the applicant(s)
is/are entitled to make the application are as follows:

12.

The applicant is the assignee of the
invention from the said actual inventors.

(Note: Paragraphs 3 and 4 apply only to Convention applications)

3. The basic application(s) for patent or similar protection on which the application is based
is/are identified by country, filing date, and basic application(s) as follows:

13.

NEW ZEALAND

24 June, 1976

N.Z. FOREST PRODUCTS LIMITED

4. The basic application(s) referred to in paragraph 3 hereof was/were the first application(s)
made in a Convention country in respect of the invention the subject of the application.

At least PLACE of

IN DATE of

IN DATE of

IN DATE of

Declared at ⁽¹⁾ Auckland
Dated ⁽²⁾ 22nd Sept 1977

⁽³⁾ *[Signature]*

To: The Commissioner of Patents

This invention relates to the recovery of useful compounds from vegetable matter and has been devised particularly to provide a method for the recovery of phenolic compounds from the bark of trees and/or shrubs for use in the manufacture of bonding, sizing and coating agents, and various resins. While the invention will be described with reference to bark it should be appreciated that this is by way of example only, and that other vegetable matter can be used.

10. The bark of trees and shrubs, although varying in composition between species contains significant amounts of phenolic materials capable of reacting with aldehydic substances, and such phenolic material or at least part thereof may be extracted for use in the manufacture of certain bonding, sizing and coating agents, and various resins. Although it is a relatively simple matter to
15. prepare aqueous phenolic extracts from bark in the laboratory the phenolic materials contained in the extracts are very reactive, and considerable difficulty is experienced in preventing or minimizing premature reactions, these premature reactions committing reaction sites which would otherwise be available, thereby making the extracts obtained less useful, and of excessively high viscosity even at low concentrations. Even as the tree or shrub grows, and also during storage of the bark and the extraction process, premature reactions are advancing, committing useful reaction sites which would otherwise be available, so that although precautions
20. can be taken, up until this time, no means has been known for preventing these premature reactions from taking place at the present time although the problems they cause are well known.
- 25.

The only satisfactory aqueous extract of bark readily available at the present time is derived from wattle (acacia) species by a



hot water extraction process. However, certain species are not grown in sufficient quantities in many regions and the hot water extraction process is not favoured for use with most bark species principally on the account of the uneconomically low yields of extractives obtained. Even when the hot water extraction method is used with preferred bark from wattle species problems caused by premature reactions still occur.

Bark is a waste product of forest industries and is available in substantial quantities but to be useful as a source of phenolic materials it is essential that an extraction method suited to the predominant bark species available in a particular region be provided.

It is therefore an object of the present invention to provide a method of treating a phenolic material from vegetable matter such as bark, such as from trees, shrubs, and the like, and for use in the manufacture of bonding, sizing and coating agents, and in various resins.

The term "an aqueous, phenolic, bark extract" as used throughout the specification and claims is hereinafter defined as an aqueous solution including at least some phenol or phenolic material, having been extracted from bark, such as for example bark from trees or shrubs.

According to this invention there is provided a method of treating an aqueous, phenolic, bark extract including:

adjusting the pH of said extract so that the pH is above pH 7;

maintaining said extract temperature above 49°C for a period of up to 90 minutes;



By way of example, the use of the invention in conjunction with the extraction of substantially phenolic material from the bark of Pinus radiata will now be described. The bark from other species including Pinus ponderosa, Pinus albertii, Pinus contorta, Pinus pseudotsugae latifolia, Eucalyptus regnans, Eucalyptus delegatensis, would provide similar examples. It will be appreciated that the process must be suited to the bark derived from species growing in a particular region and pinus radiata has been chosen because it is the predominant species growing in New Zealand. It should not be implied however that phenolic extracts can be prepared from the bark of any species of tree as some barks contain very little useful phenolic material and the economics of extracting this phenolic material from them becomes unattractive. Conversely, certain species not grown in sufficient quantity in New Zealand such as Acacia species have bark with a very high phenolic material content which can be simply extracted using hot water infusion. Extraction of Pinus radiata bark is more difficult than this however and we have found that chemical compounds must be employed when manufacturing commercial extracts from this species as described and claimed in our New Zealand patent specification No. 179933. Figure 1 is a flow diagram showing a preferred form of the present invention used in conjunction with a preferred method of extracting substantially phenolic material from bark as disclosed in our New Zealand patent specification No. 179933. The extraction method is represented by that part of the process A-B-C and the present invention by that part of the process C-D. The extraction method A-B-C is a preferred counter current method wherein an aqueous extraction solution flows in one direction (A-C) and bark particles flow in the counter direction (E-F). Water

entering the process at A tends to wash extracted phenolic material and compounds from the spent bark before the bark emerges at F so that the phenolic material and compounds are not wasted. As the water which is heated flows in the direction indicated by the arrow marked AQUEOUS SOLUTION FLOW suitable compounds are introduced at B. The compounds shown, sodium carbonate and sodium sulphite, are preferred compounds, selected to maximise yield of phenolic material extracted from the bark particles and to minimise the incidence of degradation or premature reactions which may take place during the extraction process. Other compounds may also be used however. The water entering the process at A may be naturally acidic, i.e. less than pH 7 but when the preferred compounds are introduced the pH increases as shown. Because the substantially phenolic material extracted from the bark is acidic however the pH tends to fall again as this material is extracted into the aqueous solution. When the extraction process is completed as represented by C the bark particles and aqueous solution are separated by suitable means, the bark particles continuing through the process as indicated by the arrow marked BARK PARTICLE FLOW and the aqueous solution, now containing phenolic material extracted from the bark particles being the substantially phenolic extract. The substantially phenolic extract now has a suitable base introduced to raise the pH to about pH 7 and preferably at least pH 8, sodium hydroxide as shown, being a suitable base. Other bases may also be used however. During that part of the process C-D the temperature of the substantially phenolic extract is maintained above 49°C and preferably at least 60°C, the combination of pH and temperature dismantling higher molecular weight phenolic material to lower